

## **P. Mechanical Behavior of Ceramic Materials for Heavy-Duty Diesel Engines**

*Andrew A. Wereszczak*

*Ceramic Science and Technology Group (CerSaT)*

*Oak Ridge National Laboratory*

*PO Box 2008, MS 6068,*

*Oak Ridge, TN 37831-6068*

*(865) 576-1169; fax: (865) 574-6098; e-mail: wereszczakaa@ornl.gov*

*DOE Technology Development Area Specialist: Dr. Sidney Diamond*

*(202) 586-8032; fax: (202) 586-1600; e-mail: sid.diamond@ee.doe.gov*

*ORNL Technical Advisor: D. Ray Johnson*

*(865) 576-6832; fax: (865) 574-6098; e-mail: johnsondr@ornl.gov*

---

*Contractor: Oak Ridge National Laboratory, Oak Ridge, Tennessee*  
*Prime Contract No.: DE-ACo5-00OR22725*

---

### **Objective**

- Characterize and model contact-induced damage mechanisms in ceramics and link them to microstructure and ultimately to wear performance, the optimization of machining, rolling contact fatigue, etc.
- Characterize the influence that independent parameters such as grain size, loading rate, confinement or residual stresses, temperature, and field effects have on the evolution of contact-induced damage.

### **Approach**

- Develop and use instrumented static and dynamic indentation and instrumented scratch test methods to interrogate quasi-plasticity and fracture processes in ceramics.
- Model contact-induced quasi-plasticity and fracture processes.
- Link damage mechanism activity to wear performance, machining, contact fatigue, etc.
- Develop strategies for engineering control (e.g., confinement, temperature) that promote the dominance of one mechanism (e.g., one that causes strengthening or hardening) over others.

### **Accomplishments**

- Established dynamic indentation and instrumented scratch testing facilities.
- Nearly completed indentation rate effect studies on several silicon nitride ceramics.
- Developed an ANSYS model that enables the study of indenter depth-of-penetration in a target material while considering elastic and yield properties of the indenter and target material, indenter diameter, etc.).

### **Future Direction**

- Characterize the influences that grain size and residual/confinement stress have on damage evolution.
  - Link indentation-induced damage evolution to that produced by scratch testing.
  - Add tension-compression strength/yield anisotropy effects to indentation model.
-

## **Introduction**

More ceramic components could be used in engines and transportation systems if they could be confidently used in more wear-related applications and if they could be manufactured and machined faster (i.e., more cost-effectively). This would occur if contact-induced damage mechanisms (which limit their mechanical performance or dictate their machinability) were understood, predictable, and controlled.

This project quantifies the loads and stresses associated with the initiation of quasi-plasticity and fracture processes in ceramics, using instrumented indentation and scratch testing. Understanding the competition and interaction among those processes is a critical part of this project as well. Though more traditional structural ceramics (e.g.,  $\text{Si}_3\text{N}_4$  and  $\text{ZrO}_2$ ) under consideration for diesel engine components are the primary focus, other classes of ceramics and brittle materials that could provide greater understanding of damage evolution fundamentals are also studied. These include cubic oxides and non-oxides, nanoceramics and nanocermetts, piezoelectric ceramics, and micaceous ceramics.

Through quantifying the loads or stresses that initiate quasi-plasticity and fracture, contact-induced damage mechanisms in ceramics are characterized; modeled; and linked to microstructure and ultimately to wear performance, optimization of machining, rolling contact fatigue, etc. Additionally, the influence of independent parameters such as grain size, loading rate, confinement or residual stresses, temperature, and field effects on the evolution of contact-induced damage are examined. A thorough understanding of the competition of fracture and quasi-plasticity in ceramics will enable improved and faster means of manufacturing ceramic components and surface engineering (e.g., ductile regime machining) and will maximize mechanical performance in cases when surface conditions (e.g., bending) or surface-located events (e.g., wear, impact) are service-life limiters in engine and transportation system components.

## **Approach**

FY 2004 was the first full year of this project, and numerous parallel efforts were initiated. The project has five primary elements; three represent ongoing and near-term efforts, and the other two represent longer-term efforts. The first element is to

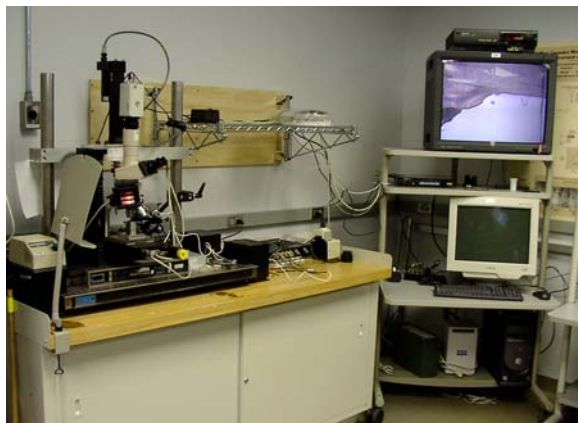
develop and use instrumented static and dynamic indentation and instrumented scratch test methods (see Figure 1) to interrogate quasi-plasticity and fracture processes in ceramics. Second, contact-induced quasi-plasticity and fracture processes are modeled. Third, ceramic materials are characterized that either are of strategic relevance to heavy-duty diesel engine components or serve as model materials for the examination of damage evolution. Fourth, damage mechanism activity is linked to wear performance, machining, contact fatigue, etc. Finally, strategies are developed for engineering control (e.g., confinement, temperature) that promote the dominance of one mechanism (e.g., one that causes strengthening or hardening) over others.

## **Results**

In order to quantify quasi-plasticity and fracture processes, instrumented static and dynamic indentation test systems were established in FY 2004, as well as instrumented scratch testing capabilities. Each of the instruments is shown in Figure 1a–c, and are all in use to experimentally characterize quasi-plasticity and fracture processes. A dedicated acoustic emission (AE) system is interfaced with all these instruments and is used to detect damage processes that result during indentation or scratch testing.

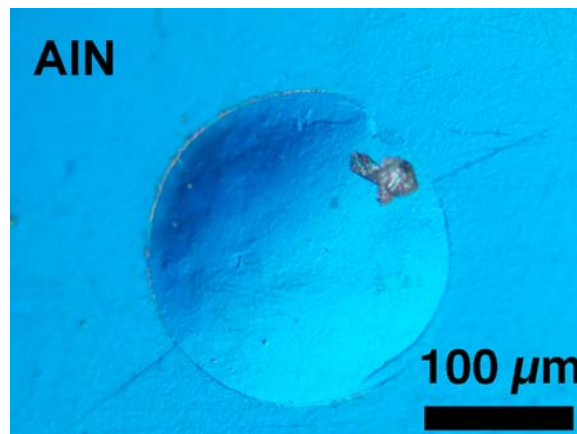
An example of a Hertzian indent is shown in Figure 2. The depth of the crater-like indent is an indication that quasi-plasticity was initiated. A circular ring crack and a median crack (2 and 8 o'clock positions) are indications that at least two different crack systems were initiated. The loads and indenter depth-of-penetration associated with the initiation of the damage mechanisms are measurable using the static instrumented indentation test system (IITS) and the dynamic indentation test systems (DITS). Their values are compared with those for other ceramics and interpreted with respect to traditionally measured mechanical properties, such as hardness, fracture toughness, and tensile strength.

AE testing and analysis was an integral part of the indentation testing because it can be efficiently used to identify the loads associated with the initiations of cracks (i.e., fracture processes). Measured load-unload histories and the loading predicted by finite element analysis (FEA) provide a reference to the AE interpretation. For example, for the spherical indent-generated ring cracks formed in the concentric pattern shown in Figure 3a, the AE spectrum



**Figure 1.** Facilities established included instrumented static (a) and dynamic (b) indentation test systems and a scratch tester (c).

(Figure 3b) shows that numerous acoustic events had occurred, with some events having greater amounts of energy than others (e.g., the event that occurred at  $\sim 175$  N). Additionally, acoustic events

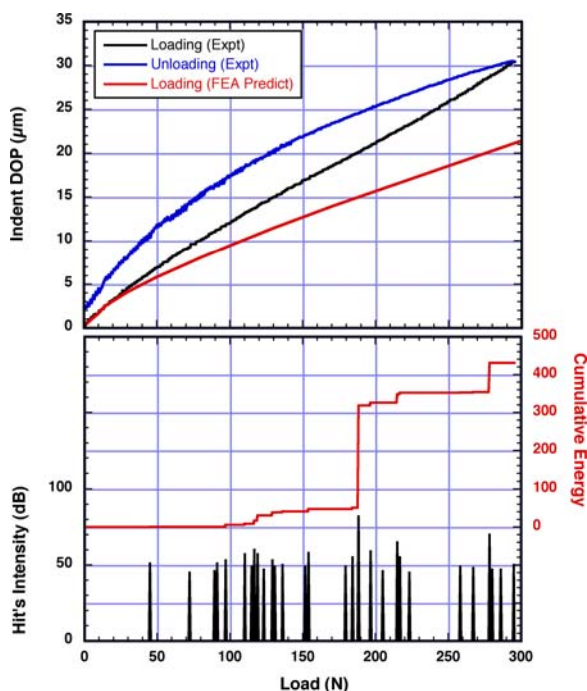
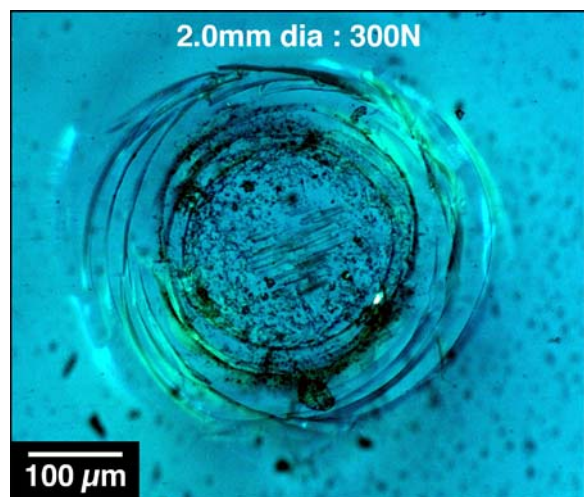


**Figure 2.** Hertzian indent illustrating quasi-plasticity (dimple) and fracture processes (ring and median crack). Indent was generated with a 2.25-mm-diam diamond indenter and 300 N maximum load.

did not occur until a load of  $\sim 48$  N, indicating that the material behaved elastically below that load. The load at which divergence of the black and red curves (i.e., where permanent damage is introduced) occurred in the indenter depth-of-penetration load curve matches fairly well with the load where acoustic signals begin to be produced, and the load where the highest-energy acoustic peak occurred matches the load on the experimental loading curve (black curve) where the slope arguably begins to increase (i.e., a relatively large increase in specimen compliance).

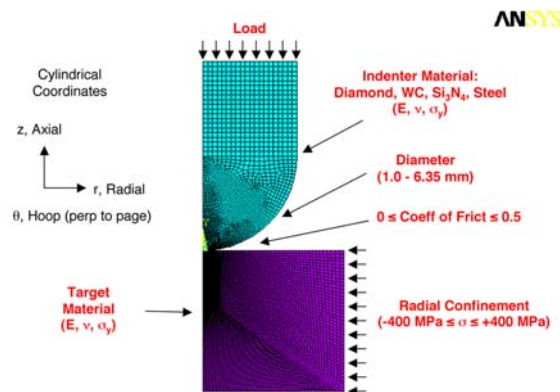
The DITS and instrumented (pendulum) scratch tester were interfaced with a high-speed data acquisition system, and experimental familiarity was gained with both testers. Gas gun settings, shape pulsing, and overall test approaches were optimized with the dynamic indenter to produce controllable low-load dynamic indentations (i.e., a few to tens of Newtons). Also, an ability to test specimens having a nominal geometry of  $3 \times 4 \times 4$  mm (a specimen cut from conventional ASTM C1161B bend bars—a geometry that is readily available for many materials in the Ceramic Science and Technology Group) was established with the scratch tester. The specimen holder that came with the instrument (which also houses two load cells for normal and thrust force measurements) was designed to test specimens having the geometry of a  $\frac{1}{4}$ -in. (6.35-mm) cube—an inconvenient size.

An indentation model was developed using ANSYS FEA software that predicts the stress state

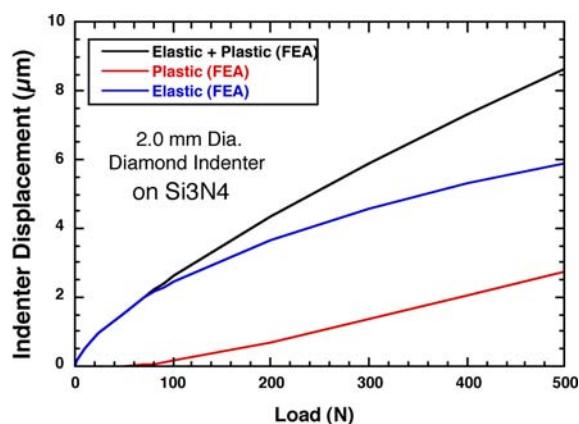


**Figure 3.** Hertzian indent of a soda lime glass (a), and (b) its measured load-unload waveform compared with the predicted loading waveform and the acoustic emission history associated with the generated crack pattern. The indent was generated with a 2.0-mm-diam diamond indenter and 300 N maximum load.

in a material subjected to Hertzian (spherical) indentation. An illustration of the model is shown in Figure 4 with a predicted response shown in Figure 5. Independent parameters that may be considered in the model are elastic properties and hardness (i.e., yielding) of the target or indenter material, indenter diameter, coefficient of friction, confinement pressure, and, of course, applied indenter load. In-



**Figure 4.** ANSYS Hertzian indentation model.

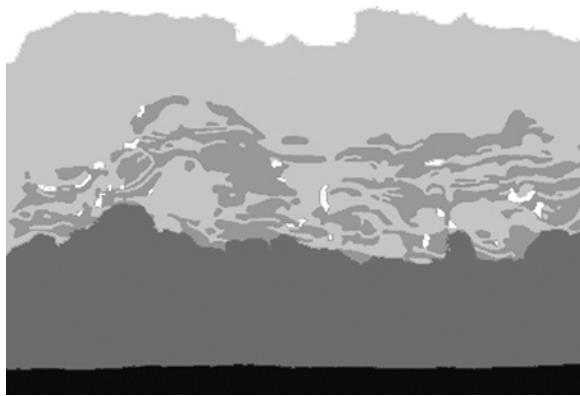


**Figure 5.** Example of predicted indentation response. The total indent-depth-of-penetration (top curve) is the sum of elastic (middle curve) and permanent deformation (bottom curve).

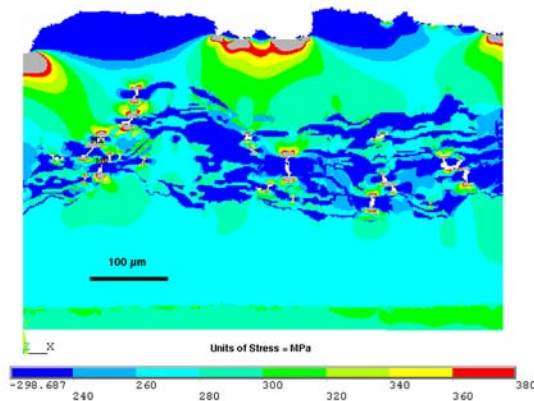
terpretation of the von Mises and First Principal tensile stresses is enabling the study of the competition between plasticity and fracture processes in the test material (linkable to machining and wear phenomena), and the results are being correlated to experimental observation.

$\mu$ -FEA, a microstructure-based FEA computer program, was developed and its copyrighting is under way through ORNL's Technology Transfer and Economic Development organization.  $\mu$ -FEA is a graphics-user-interfaced LabVIEW executable program that serves as a pre- and post-processor to the ANSYS FEA solver, and it enables stress analyses of both real (e.g., imaged with scanning electron microscopy or optical microscopy) and simulated material microstructures. An example of an analyzed microstructure and its subsequent residual stress calculation is shown in Figures 6a and b. For one of its applications,  $\mu$ -FEA is being used to study stress concentrators, deformation, and microcracking





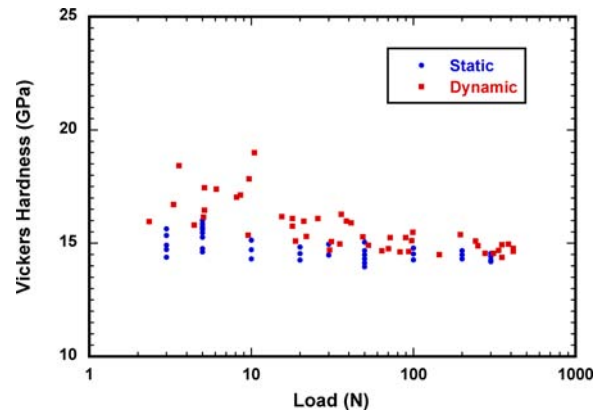
ANSYS



**Figure 6.** Digital microstructure of (a) coating and (b)  $\mu$ -FEA residual stress evaluation induced from thermal expansion mismatches between material constituents.

around pores and large grains and in secondary phases in materials subjected to a Hertzian contact stress gradient.

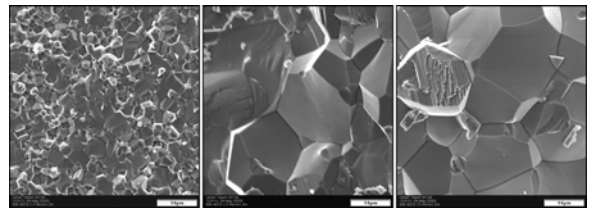
Silicon nitride specimens of Ceralloy 147-31N, NBD200, SN101C, NC132, and TSN-03NH were sectioned and either underwent or are undergoing instrumented static and dynamic indentation and instrumented scratch testing. An apparent rate dependence (i.e., quasi-plasticity rate dependence) on Vickers Hardness for Ceralloy 147-31N is shown in Figure 7. There is a rationale behind the choice of testing each silicon nitride composition. The 147-31N grade is presently used as a cam roller follower material, so interest exists in quantifying its indentation and scratch responses. NBD200, SN101C, and TSN-03NH are ball bearing grades, so their testing is relevant to the examination of rolling contact fatigue behavior. NBD200 is a NIST standard reference material for Knoop hardness, and NC132 is a



**Figure 7.** Static and dynamic Vickers hardness of Ceralloy 147-31N as a function of indentation rate.

NIST standard reference material for fracture toughness, so they serve as references for performance comparison among all the silicon nitrides.

To examine the effect of grain size on damage due to cracking and quasi-plastic yielding generated with instrumented indentation and scratch testing, three different mean-grain-sized (2–3, 15, and 25  $\mu$ m) aluminas were acquired (Ceradyne, Costa Mesa, CA). Microstructures of the three materials are shown in Figure 8. The alumina has 99.9% purity, has magnesium as a sintering aid, and has relatively narrow size distributions. Alumina was chosen as the model material in which to examine grain size effects because of the ease associated with measuring residual stresses on it using optical fluorescence, and because of the immediate availability of such an analytical tool. Residual stresses will be measured in, around, and under generated indents and scratches, and the results will likely prove to be valuable for interpreting grain size effects on cracking and quasi-plasticity.



**Figure 8.** Microstructures of 99.9% alumina with average grain sizes of (left) 2–3 microns, (middle) 15 microns, and (right) 25 microns.

The effects of confinement (i.e., pressure) on instrumented static and dynamic indentation, instrumented scratch testing, and the competition of

fracture and quasi-plastic damage mechanism behaviors are under examination. The 99.9% alumina is being machined, and metal sleeves/tubes will be shrink-fitted about it to produce a radial confinement. The residual stress field on the surface of the radially confined alumina will be quantified using optical fluorescence; and the static, dynamic, and instrumented scratch testing will commence on it. Because the aluminas described will also serve as the model materials for this examination, grain size will also be an independent parameter in this confinement effect study. If confinement introduces beneficial effects related to damage associated with indentation or scratch testing, then the characterization of that effect will lead to its implementation into grinding or wear applications.

Experimental strategies are being pursued to enable the study of the effect of temperature on instrumented static and dynamic indentation and instrumented scratch testing. The characterization of this effect will have a direct link to optimizing heat-assisted machining of ceramic materials or promoting minimized surface damage that will result in improved wear resistance and strength.

## **Conclusions**

Static and dynamic indentation and instrumented scratch testing facilities were established to quantify the loads and stresses associated with the initiation of quasi-plasticity and fracture processes. Numerous silicon nitride ceramics are under investigation, and rate effects on hardness (i.e., quasi-plasticity) are evident. An ANSYS model was developed that enables the study of indenter depth-of-penetration in a target material while considering elastic and yield properties of the indenter and target material, indenter diameter, etc. A microstructure-based FEA software ( $\mu$ -FEA) was developed to enable stress analysis characterization at the microstructural scale and to help with the interpretation of quasi-plastic and fracture processes.

FY 2004 progress provides a nice springboard for planned FY 2005 work. The influences that grain size and residual/confinement stress have on damage evolution will be characterized. Indentation-induced damage evolution produced by scratch testing will

be linked to that generated from static and dynamic indentation. Tension-compression strength/yield anisotropy effects will be added to the indentation model and will enable the study of how (or if) hardness is linked to quasi-plasticity and how (or if) tensile strength and fracture toughness are linked to fracture processes.

## **Publications/Presentations**

“Evaluation of Ceramic Deformation Processes Through Hertzian Indentation,” presented at the Tank and Automotive Research Development and Engineering Center-ORNL-Army Research Laboratory Advanced Materials Meeting, Network Computing Services, Minneapolis, February 5, 2004.

“ORNL Characterization of Ceramics for Armor, Transportation, and Energy Applications,” presented at Cercom, Inc., Vista, CA, February 25, 2004.

“ORNL Characterization of Ceramics for Armor, Transportation, and Energy Applications,” presented at Ceradyne Inc, Costa Mesa, CA, February 26, 2004.

“Evaluation of Ceramic Deformation Processes Through Instrumented Hertzian Indentation,” presented at 2004 Interagency Coordinating Committee on Structural Ceramics, National Science Foundation, Arlington, VA, April 14, 2004.

“Evaluation of Ceramic Deformation Processes Through Hertzian Indentation,” presented at Saint-Gobain, Worcester, MA, August 3, 2004..

## **Copyrights**

$\mu$ -FEA, a microstructure-based finite element analysis computer program, was developed and is undergoing copyrighting through ORNL's Technology Transfer and Economic Development organization.  $\mu$ -FEA is a graphics-user-interfaced LabVIEW™ executable program that serves as a pre- and post-processor to the ANSYS finite element analysis solver and enables stress analyses of both real (e.g., imaged with scanning electron microscopy or optical microscopy) and simulated material microstructures.